PROJECT NUMBER : 4016
PROJECT TITLE : Paper Technology
PROJECT LEADER : S. D. Baldwin
PERIOD COVERED : December, 1990

PAPERMAKING PROCESS DEVELOPMENT (B. Rogers)

I. EXPERIMENTAL PAPERS (B. Rogers, N. Gautam)

A. **Objective:** Develop proprietary cigarette papers for low sidestream smoke or product improvement.

B. **Results:** Due to the current backlog of handsheets for the Low Sidestream project, a number of requests were transferred to The University of Maine. Handsheet procedures were developed at Maine that duplicate the methods used at Philip Morris. A total of six handsheet requests were completed during the first week of production. All sheets will be coated at Philip Morris.

Handsheet facilities are scheduled to be moved during the first week of January to Semiworks. In addition, a second 12" square handsheet mold has been ordered to facilitate handsheet production. The new lab should be fully operational by the second week of January.

Three areas of process development are being pursued at the University of Maine to improve both sheet appearance and strength. The object is to develop the capability to further reduce the fiber length of flax at equal freeness. Two new types of flax have been obtained (Kimberly-Clark and Type B flax from Danforth) for evaluation early next year. The condition of the beater at Maine will also be evaluated in January to determine if this unit's refining capabilities have deteriorated. New disc refining plates have been ordered.

C. **Plans:** Evaluate process changes at University of Maine. Install second handsheet mold in new laboratory facilities.

II. BANDED PAPERS (B. Rogers, N. Gautam, J. Ryder, S. Baldwin)

A. **Objective:** Incorporate cross directional bands of fiber and/or filler in cigarette papers in order to vary cigarette burn rate.

B. **Results:** Papers were produced with 5 g/m^2 Cellulon applied in the banded regions. Partial bands, bands composed of spots, and spotted papers were produced, all with an inherent porosity <1 Coresta in the banded region. Bands composed of <5 g/m^2 Buckeye Cellulose expanded fibers were ineffective in modifying burn rate since the banded section's porosity was 5 Coresta.

Quantities of Cellulon were cleaned by an experimental process in an effort to make this material more suitable for use. Samples have been submitted for nitrogen and

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NMR analyses and to make banded handheets. The color of the cleaned material is much whiter, and the material has no malodor, as the raw Cellulon has.

Extrusion technology was discussed with Nordson as a possible method of applying cross directional bands to either wet (wet end of a paper machine) or dry paper. The technology is currently available to apply cross direction bands on dry paper up to 16" wide at about 100 fpm (single nozzle). A short nozzle section was ordered and will be evaluated on paper (both wet and dry) with cellulosic containing slurries (Cellulon and Buckeye's expanded fiber).

One of the banded drums at Maine will be modified for use as a "rotogravure" type cylinder to evaluate this concept. The inside of the drum will be blanked off and a portion of the slots will be filled to reduce the volume. Maine will construct a slurry box and doctor blade that will be mounted on the outside of the drum. This apparatus should allow the application of bands with ~1 g/m^2 add-on.

Beloit Corporation's paper machine research facility was toured to evaluate their ability to scale up the Daubing Dandy concept. They appear be a viable alternative to developing and testing the concept at either Philip Morris or another contractual research facility. Several possible designs options were discussed. Beloit will prepare a list with cost and completion dates for each option by the middle of January.

The sheet properties resulting from use of a top wire drainage device for banded papers was modeled in the handsheet lab by constructing bilayer handsheets. The moisture level of the banded region was varied (6 to 48% solids) while maintaining the base sheet at a constant solids level (flax/carbonate sheet at 12.8% solids). Sheet bonding decreased and both porosity and caliper increased as the solids level of the banded region was increased. Apparently formation and drainage of the banded material on the handsheet mold increased this fraction's porosity. The increase in caliper and decrease in tensile is an indication that the banded region is not being incorporated into the base sheet.

III. FILTER DEVELOPMENT SUPPORT (B. Rogers, N. Gautam)

A. **Objective:** Support the development of a proprietary filter material in web form for utilization by Filter Technology.

B. **Results:** The success of the CA/softwood and 100% softwood models is attributed to the long cellulosic fiber content a portion of which tends to remain intact during the filter making's corrugation process. All the sheets lint excessively which is attributed to the use of CMC on the Yankee (to adhere the sheet for creping). Future runs, scheduled for early January, will be directed at improving the strength of the CA/softwood model and reducing the degree of lint.

Additional filter webs were analyzed for surface area. Combinations of partially acetylated cellulose and Cellulon give the highest surface area (~2.2 m^2/g, above CA web at ~2.0 m^2/g, standard deviation = 0.01 m^2/g). Handsheets were also prepared

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with partially acetylated Cellulon, partially acetylated softwood and Cellulon for surface area analysis.

C. Plans: Complete scheduled web runs at James River.

PAPER PRODUCT DEVELOPMENT (B. Goodman)

I. REDUCED SIDESTREAM CIGARETTES (B. Goodman, B. Floyd, S. Baldwin)

A. Objective: Develop subjectively acceptable cigarettes with reduced sidestream.

B. Results:

Trim: Kimberly-Clark made a millrun of base papers at three different porosities, each with ~10.5% monopotassium phosphate added at their Ancram mill. Porosities were acceptable and phosphate levels were very close to target (~10% by our colorimetric analysis). Cigarettes were made in Louisville with papers at 11, 13, and 14.5 Coresta, measured with the 20 cm2 clamp. The ventilation target of 66% was hit with the 0.25 cm pressure drop tipping paper suggested by Cigarette Development. Because of this, tar deliveries were lowered compared to the November run. Sidestream reductions ranged from 69 to 74%, and the highest tar delivery was 6.6 mg. Cigarettes from the December 11 Louisville run were screened for weight, RTD, and dilution and given to Cigarette and Flavor Development for subjective smoking. Test 2, the 12 - 13 Coresta paper with 10.4% phosphate was chosen as the preferred model. Test 2, the preferred model, gave 71% reduction. From these limited data, we are achieving a 70% visibility reduction at a 95% confidence level, There were no observed differences in the number of cigarettes going out (22%), and there was no staining behind the charline.

The 11 experimental Superslims models from the November run in Louisville were used to determine the effect of porosity, calcium carbonate level and phosphate level on visibility, tar and puff count. Good fits were obtained in each case. The most striking finding was that tar is directly related to calcium carbonate level.

C. Plans: Return to Louisville to repeat the experiment using the additional bobbins of paper still on hand. That study will be designed in collaboration with John Tindall.

PAPER AND CELLULOSE DEVELOPMENTAL STUDIES (G. Bokelman, S. Tafur, S. Baldwin)

I. MAGNESIUM CARBONATE PAPERS (G. Bokelman, S. Tafur)

A. Objective: To produce commercial quantities of a magnesium carbonate paper.

B. Results: The first step of a two-step process developed for the grinding of Baymag magnesite was executed. The first step, involving impaction crushing from rocks to a coarse sand, was completed on 45 tons of magnesite at Industrial Crushing (Cambridge, Ohio). Prior to initiation of this work the plant lay out and the material

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composition of all relevant equipment were evaluated to help assure that there would be no contamination problems. In addition, an analyzed, compatible mineral was processed through this equipment just prior to the crushing of the magnesite to evaluate any potential for contamination. Nine tons of the crushed Baymag magnesite, representing two different particle size ranges, have been shipped to Midwest Custom Services, Inc. (Fort Wayne, Indiana) for the second step in the grinding process. The magnesite crushed at Industrial crushing in mid December was processed on both the pin mill (with tungsten-carbide liner) and in the jet mill (with composite tungsten-carbide liner). These magnesite samples were processed on the commercial scale equipment at no cost to Philip Morris, and will be analyzed for trace metal content prior to processing larger quantites of the material to be used for a mill run at Ecusta.

Analyses were obtained for K, Mg and Ca levels of the Ecusta mixed filler handsheets sized with K_3 citrate/citric acid. Levels of solublized Mg and Ca were found in a water wash of the paper to suggest the citric acid readily reacted with the $Mg(OH)_2$ and, to a lesser extent, the $CaCO_3$ filler in the paper to form soluble citrates.

C. Plans: Pending the results of the trace metal analyses, preparation of larger quantities of the ground magnesite is scheduled for the week of January 22.

II. MECHANISTIC STUDIES (S. Tafur, G. Bokelman, S. Baldwin)

A. Objective: To conduct research to elucidate mechanisms by which low sidestream papers act and to develop support for patent claims.

B. Results: Samples of magnesite/$CaCO_3$/$Mg(OH)_2$ handsheets from Ecusta sized with K_3 citrate/citric acid have been submitted for analyses to determine if soluble and insoluble forms of magnesium can be differentiated. Probable reaction of the citric acid with the $Mg(OH)_2$ filler to form magnesium citrate may contribute to the improved subjectives.

The results of a Box-Behnken study conducted with the Solar Energy Research Institute (SERI) quantified the effect of potassium ion concentration, pH, and pyrolysis temperature on the cellulose and paper pyrolysis product slate. Differences in the product mix resulting from different additives to papers are believed to affect subjectives. The work was done using Molecular Beam Mass Spectrometry (MBMS) to evaluate the pyrolysis and combustion product mix for flax samples and high basis weight paper samples to which three levels of KH_2PO_4 were added at three pH levels (3, 4.4, and 9). The pH 9 represents K_2HPO_4. Results showed that three main product slates were a direct function of pH, potassium ion concentration, and temperature.[1] Those three principal classes of compounds were anhydrosugars, aldehyde and carbonyl compounds, and furan-type compounds. The data generated to date by this work is considered by patent counsel to be sufficient to support our patent claims for PM 1393. This patent is for the high paper which will be used for the single wrap Virginia Slims Superslims. Additional data also indicates that there

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is an interaction between the phosphate and the calcium carbonate which affects the products formed when the paper is burned or pyrolyzed. The effect depends on the amount of oxygen present.

Other parallel experiments on a bacterial cellulose grown on ^{13}C-enriched media have now shown definitive results. Acetobacter xylinum was grown on media that included glucose that was 13C labelled at the C-1 position, resulting in 13C levels of approximately 14%. This cellulose was pyrolyzed under both slow- and fast-heating conditions and the mass spectral results compared to unlabeled cellulose that was prepared in the same fashion. In the labelled sample, a higher peak was observed at m/z 145, which indicates inclusion of the C-1 carbon into levoglucosan. No enhancement was observed for m/z 61, which indicates that the glycolaldehyde does not come from the C-1 carbon in the anhydroglucose ring. Addition of 0.1% potassium as KOH did not yield any enhancement of m/z 61. Again this indicates that the mechanism of formation of glycolaldehyde does not involve incorporation of the C-1 carbon. This is important mechanistic information concerning the mechanism of action of alkali metals on cellulose and paper pyrolysis and combustion[1,2].

C. Plans: Evaluate the effect of KH_2PO_4 on cellulose pyrolysis using the labelled material. Evaluate the effect of oxygen content on products of paper and cellulose pyrolysis and combustion.

D. References:

1. R. Evans, F. Abglevor, H. Chum, D. Chadick, J. Wooten, S. Baldwin, "On the Mechanism of Pyrolysis of Cellulose to Low Molecular Weight Compounds", paper presented at ACS National Meeting, Washington, D.C., August 29, 1990.

2. R. Evans, F. Abglevor, H. Chum, D. Chadick, J. Wooten, S. Baldwin, "New Approaches to the Study of Cellulose Pyrolysis", paper to be presented at ACS National Meeting, April, 1991.

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